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## Inclusion compounds of a borneol dumb-bell host with methylcyclohexanones and 2-butanols: Structures and resolutions

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The host 2,2'-(benzene-1,4-diyl-diethynylene) diborneol has been employed to resolve racemic methylcyclohexanones and 2-butanol. For 2-methylcyclohexanone, the resultant inclusion compound yielded an enantiomeric excess of 72% (*S*) while with 3-methylcyclohexanone the enantiomeric excess was 57% (*S*). The host failed to resolve (*R,S*)-2-butanol, and the inclusion compounds derived from the (*R,S*)-, (*R*)- and (*S*)-2-butanol are isostructural, being dominated by a stable framework of host•••host hydrogen bonds. The non-templating effect of the 2-butanol was explained in terms of the secondary interactions occurring in these structures which were also analysed by the program CrystalExplorer.

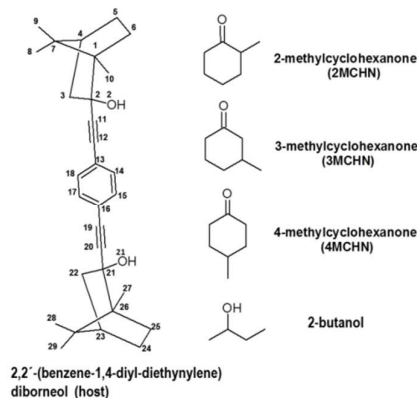
### Introduction

Functionality and bulkiness are structural criteria typical of host molecules designed for guest separation.<sup>1</sup> Molecules meeting these requirements have been developed in a variety of geometric structures<sup>2</sup> including those resembling the shape of a wheel-and-axle<sup>3</sup> or a dumb-bell.<sup>4</sup> Usually, the rigid linear axle of the dumb-bell molecule features a sequence of ethynylene or 1,4-phenylene units or a combination of both while the ball-shaped terminal stoppers are bulky hydrocarbons such as adamantyl groups.<sup>5</sup> Owing to their particular structure, the dumb-bell-shaped hosts pack inefficiently leaving channels or voids that can accommodate organic guests of suitable size and chemical affinity.<sup>4,5</sup> In a special kind of purpose-built host structures of the dumb-bell kind, chiral borneol moieties were employed as bulky terminal substituent groups in order to generate enantioseparation of guest molecules.<sup>6</sup>

A typical example of this molecular design is the host (**H**), 2,2'-(benzene-1,4-diyl-diethynylene)diborneol, specified in Scheme 1. The ability of this host compound to include organic guests has been investigated before<sup>7</sup> and some preliminary results for enantioseparation of chiral guest species have been reported.<sup>6a</sup> Nonetheless, challenging tasks with regard to the exploitation of this chiral host compound as a resolving agent

are continuing.

We present herein the structures of the inclusion compounds obtained with the title host and 2-methylcyclohexanone; **H·(2MCHN)** (**1**), 3-methylcyclohexanone; **H·(3MCHN)** (**2**), 4-methylcyclohexanone; **H·(4MCHN)** (**3**), (*S*)-2-butanol; **3H·2((*S*)-2-BuOH)** (**4**), (*R*)-2-butanol; **3H·2((*R*)-2-BuOH)** (**5**) and (*RS*)-2-butanol; **3H·2((*RS*)-2-BuOH)** (**6**) (Scheme 1). The borneol moiety has three stereogenic centres at C1(*R*), C2(*R*) and C4(*R*) as a consequence of the natural D-(+)-camphor from which it was derived. The configuration at the three stereogenic centres was also confirmed by structure determination of the inclusion compounds containing the host compound and either (*R*)-2-butanol or (*S*)-2-butanol as an internal chiral reference. With the aid of Hirshfeld surface analysis, we analyse the packing in the structures and rationalise the results of the resolution experiments in terms of non-bonded interactions.



Scheme 1 Structures of host and guest compounds used in this study

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Table 1 Selected crystal data for structures 1-6

Compound No	1	2	3	4	5	6
Code	H-(2MCHN)	H-(3MCHN)	H-2(4MCHN)	3H-2((S)- 2-BuOH)	3H-2((R)- 2-BuOH)	3H-2((RS)- 2-BuOH)
Host: Guest	1:1	1:1	1:2	3:2	3:2	3:2
Formula weight	542.77	542.77	654.98	1440.05	1440.05	1440.05
Structure formula	C <sub>37</sub> H <sub>50</sub> O <sub>3</sub>	C <sub>37</sub> H <sub>50</sub> O <sub>3</sub>	C <sub>44</sub> H <sub>62</sub> O <sub>4</sub>	C <sub>98</sub> H <sub>134</sub> O <sub>8</sub>	C <sub>98</sub> H <sub>134</sub> O <sub>8</sub>	C <sub>98</sub> H <sub>134</sub> O <sub>8</sub>
Space group	P2 <sub>1</sub> 2 <sub>1</sub> 2 <sub>1</sub>	P2 <sub>1</sub> 2 <sub>1</sub> 2 <sub>1</sub>	P1	P2 <sub>1</sub> 2 <sub>1</sub> 2 <sub>1</sub>	P2 <sub>1</sub> 2 <sub>1</sub> 2 <sub>1</sub>	P2 <sub>1</sub> 2 <sub>1</sub> 2 <sub>1</sub>
<i>a</i> (Å)	11.7327(2)	11.719(2)	6.5774(1)	11.9564(4)	11.7623(6)	11.8542(4)
<i>b</i> (Å)	17.388(2)	17.993(4)	10.920(2)	12.0231(4)	12.0280(6)	12.0125(4)
<i>c</i> (Å)	31.903(4)	30.484(6)	13.999(3)	60.7918(2)	61.710(3)	61.234(2)
$\alpha$ (°)	90	90	84.03(3)	90	90	90
$\beta$ (°)	90	90	81.51(3)	90	90	90
$\gamma$ (°)	90	90	77.91(3)	90	90	90
Volume (Å <sup>3</sup> )	6508.5(14)	6428(2)	969.7(3)	8739	8730.6(8)	8719.6(5)
<i>Z</i>	8	8	1	4	4	4
<i>D</i> <sub>c</sub> /g cm <sup>-3</sup>	1.083	1.122	1.122	1.095	1.096	1.096
$\mu$ (mm <sup>-1</sup> )	0.067	0.069	0.070	0.067	0.067	0.067
<i>F</i> (000)	2272	2368	358	3144	3144	3140
Goodness of fit on <i>F</i> <sup>2</sup>	0.981	1.030	1.022	1.029	1.026	1.021
<i>R</i> <sub>1</sub> / <i>wR</i> <sub>2</sub> [ <i>I</i> > 2 $\sigma$ ( <i>I</i> )]	<i>R</i> <sub>1</sub> = 0.0848 <i>wR</i> <sub>2</sub> = 0.2303	<i>R</i> <sub>1</sub> = 0.0536 <i>wR</i> <sub>2</sub> = 0.1340	<i>R</i> <sub>1</sub> = 0.0419 <i>wR</i> <sub>2</sub> = 0.0863	<i>R</i> <sub>1</sub> = 0.0769 <i>wR</i> <sub>2</sub> = 0.1961	<i>R</i> <sub>1</sub> = 0.0676 <i>wR</i> <sub>2</sub> = 0.1643	<i>R</i> <sub>1</sub> = 0.0637 <i>wR</i> <sub>2</sub> = 0.1384
<i>R</i> <sub>1</sub> / <i>wR</i> <sub>2</sub> (all data)	<i>R</i> <sub>1</sub> = 0.1324 <i>wR</i> <sub>2</sub> = 0.2666	<i>R</i> <sub>1</sub> = 0.0676 <i>wR</i> <sub>2</sub> = 0.1432	<i>R</i> <sub>1</sub> = 0.0419 <i>wR</i> <sub>2</sub> = 0.0863	<i>R</i> <sub>1</sub> = 0.1282 <i>wR</i> <sub>2</sub> = 0.2276	<i>R</i> <sub>1</sub> = 0.1088 <i>wR</i> <sub>2</sub> = 0.1888	<i>R</i> <sub>1</sub> = 0.1167 <i>wR</i> <sub>2</sub> = 0.1616

## Experimental

All chemicals were purchased from Sigma Aldrich and used without further purification. Single crystals of compounds **1-6** were prepared by dissolving the host compound in the appropriate guest and allowing the solvent to evaporate slowly. Colourless crystals were obtained after a few days.

## X-ray Crystallography

Intensity data for compound **1**, **2**, **4**, **5** and **6** were collected on a Bruker DUO APEX II diffractometer<sup>8</sup> with graphite-monochromated MoK $\alpha$  radiation ( $\lambda = 0.71073$  Å) at 173K using an Oxford Cryostream 700. Data collection and cell refinement were performed using SAINT-Plus<sup>9</sup> and the space groups were determined from systematic absences using XPREP<sup>10</sup> and further justified by the refinement results. Accurate unit cell parameters were refined on all data. Intensity data for compound **3** were collected on the Nonius Kappa CCD single crystal X-ray diffractometer using graphite monochromated MoK $\alpha$  radiation ( $\lambda = 0.7107$  Å,  $T = 173$  K) generated by a Nonius FR590 generator at 50 kV and 30 mV. The intensity data were collected by the standard  $\phi$  and  $\omega$  scan techniques. The strategy for data collection was evaluated using COLLECT<sup>11</sup> software. Integration and scaling were performed using (DENZO and SCALEPACK).<sup>12</sup> The structures were solved using SHELXS-97<sup>13</sup> and refined using full-matrix least squares methods in SHELXL-97, within the X-Seed<sup>14</sup> graphical user interface. Non hydrogen atoms of the host compounds were refined anisotropically, while those belonging to the guest, were refined isotropically due to

disorder. Except in structure **2**, Hydroxyl hydrogen atoms were located in the difference electron density map and were fixed to calculated distances derived from the distance-dependent neutron-normalised method.<sup>15</sup> Hydroxyl hydrogen atoms in structure **2** could not be located in the difference electron density map and were placed at calculated positions. The hydrogen atoms bound to carbon atoms were placed at idealized positions and refined as riding atoms. Crystal data are given in Table 1 and hydrogen bonding details are given in Table 2. Crystallographic data for the structures have been deposited with the Cambridge Crystallographic Centre CCDC 1054466-1054471. Copies of this data can be obtained free of charge from the director, CCDC, 12 Union road, Cambridge, CB2 1EZ (deposit@ccdc.cam.ac.uk).

## Results and discussion

Inclusion compound **1**, H-(2MCHN), derived from racemic 2-methylcyclohexanone crystallises in the orthorhombic space group P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, *Z*=8, there are thus two hosts and two guest molecules in the asymmetric unit. The two guest molecules, labelled A and B, display disorder (Fig. 1(a)). Guest A was refined as two moieties with site occupancy factors of 69/31%, both with (S)- configuration and guest B was refined with site occupancy factors 72% (S)- and 28% (R)- configurations. Thus the overall enantiomeric excess in this structure is 72% (S). The structure is stabilised by (host)-O-H...O(H)-(host) and (host)-O-H...O=C-(guest) hydrogen bonds and the important features of the packing are the channels running along [100] in which the guest molecules are located, as shown in Fig. 1(b).

Compound **2**, H-(3MCHN) was grown from a solution of the host compound in 3-methylcyclohexanone and crystallises in

the orthorhombic space group  $P2_12_12_1$ ,  $Z=8$ , with two host and two guest molecules in the asymmetric unit. The guest molecules have once again been labelled A and B as shown in Fig. 2. The guest molecule A was modelled as (*S*)-3-methylcyclohexanone at full occupancy while guest B is disordered over two positions with 57% (*S*)- and 43% (*R*)-configuration. The molecules in structure **2** pack in a similar fashion as structure **1** (Fig. 1(b)) with the guest molecules located in channels along the *a* axis. The structure is stabilised by (host)-O-H...O=C-(guest) as well as (host)-O-H...O-H-(host) hydrogen bonds.

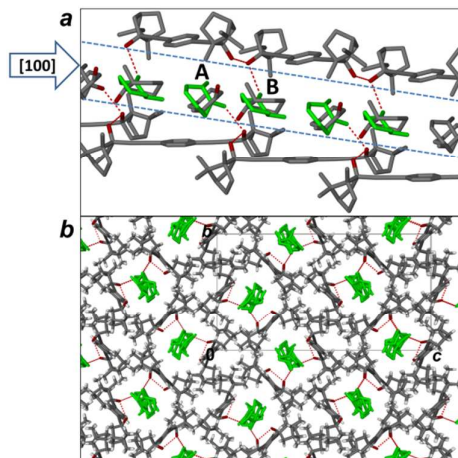


Fig. 1 (a) The molecular structure of **1** showing hydrogen bonding between the host and the guest molecules and (b) packing showing channels of guest molecules along [100]

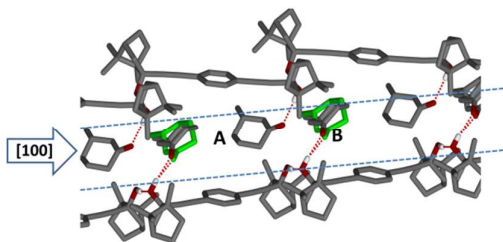


Fig. 2 The molecular structure of **2** showing hydrogen bonding interactions between the host and guest molecules. Guest A is disordered over two positions of (*R*) and (*S*) configuration while guest B is (*S*) configuration at full occupancy

Compound **3**, **H·2(4MCHN)**, was grown by slow evaporation of a 4-methylcyclohexanone solution of the host. The compound crystallises in the triclinic space group  $P1$ ,  $Z=1$ , with two guest and one host molecule in the asymmetric unit (Fig. 3(a)). Fig. 3(b) shows the packing diagram of **3** with guest molecules located in spaces between the host molecules parallel to the *bc* plane. The guests interact with the host via (host)-O-H...O=C-(guest) interactions. Hydrogen bond details for structures **1-3** are given in Table 2.

Compounds **4-6**, **3H·2((*S*)- 2-BuOH)**, **3H·2((*R*)- 2-BuOH)** and **(3H·2((*RS*)- 2-BuOH))** were grown by slow evaporation of solutions of the host in (*S*)-, (*R*)- and (*RS*)-2-butanol respectively. The three compounds crystallise in the same

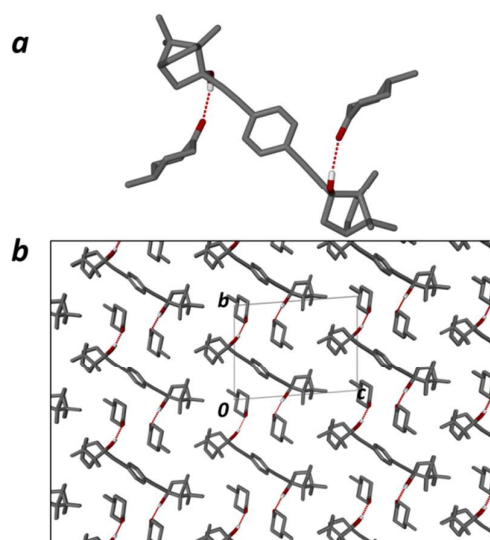


Fig. 3 (a) The asymmetric unit and (b) the packing diagram of **3** as viewed along the *a* axis. The guest molecules are located in spaces between the host molecules

orthorhombic space group  $P2_12_12_1$ ,  $Z=4$ , with similar unit cell parameters. Their asymmetric units consist of three host and two guest molecules and the packing is dominated by cross linked double chains of host molecules, running along [010], shown in Fig. 4. The three independent host molecules are coloured cyan (host A), green (host B) and red (host C) and the guests are coloured lavender (guest 1) and dark blue (guest 2). The structures consist of hydrogen bonded tetramers comprising three (host)-O-H...O(H)-(host) and one (host)-O-H...O=C-(guest) hydrogen bonds. In the packing both host A and host B are hydrogen bonded to themselves as well as to guest 1 and guest 2 respectively. Host C forms a bridge between the other two host networks thus completing a hydrogen bonded tetramer. The solvent accessible surface as well as the contact surface mapped using the void function in Mercury<sup>16</sup> (probe radius 1.4 Å) indicates the presence of two types of cavities in the structures. Guest 1 is located in discrete voids whereas guest 2 is located in channels down the *a* axis. In structure **4** the void space and the contact surface comprises 2.2% and 11.8% of the unit cell volume respectively. This result is repeated in structures **5** and **6** (see ESI). In structure **6** the two guest molecules are each disordered over two positions. The disorder at each position was modelled as partially populated (*R*)- and (*S*)- 2-butanol with equal occupancies. Hydrogen bond details for structures **4-6** are given in Table 2

### Hirshfeld surface analysis

In addition to conventional packing analysis, CrystalExplorer<sup>17</sup> was employed to further analyse structures **1** and **2** as well as **4-6**. Fingerprint plots were generated for guest B in structure **1** and **2** (See ESI, Fig S1).

Table 2 Hydrogen bond details for structures 1-6

D-H...A	D-H (Å)	H...A (Å)	D...A (Å)	∠D-H...A (°)	Symmetry codes
<b>H-(2MCHN)</b>					
O2-H2...O21	0.94	2.01	2.795(4)	154.3	x-1, y, z
O21-H21...O61	0.95	1.89	2.705(3)	160.1	
O31-H31...O76	0.83	2.13	2.963(4)	172.1	x+1, y, z
O51-H51...O81	0.84	1.97	2.782(4)	162.5	x-1, y, z
<b>H-(3MCHN)</b>					
O21-H21...O1	0.89	1.95	2.831(2)	168.5	x+1, y, z
O32-H32...O63	0.87	1.91	2.772(2)	172.6	
O52-H52...O32	0.86	2.01	2.829(2)	157.3	x+1, y, z
O1-H1...O77	0.96	1.87	2.811(2)	166.9	-x+1, y-1/2, -z+1/2
O1-H1...O70	0.96	1.88	2.822(4)	167.2	-x+1, y-1/2, -z+1/2
<b>H-2(4MCHN)</b>					
O2-H2...O38	0.86	2.03	2.866(2)	163.0	-x+1, y-1/2, -z+1/2
O45-H45...O31	0.81	2.09	2.901(2)	172.0	x+1, y-1, z
<b>3H-2((S)- 2-BuOH)</b>					
O2-H2...O32	0.91	2.00	2.862(3)	156.9	
O21-H21...O2	0.93	1.91	2.802(3)	161.3	x, y-1, z
O32-H32...O95	0.95	1.86	2.733(4)	152.1	x, y+1, z
O51-H51...O81	0.92	2.04	2.841(3)	145.2	
O62-H62...O91	0.94	1.85	2.766(4)	165.2	x, y-1, z
O81-H81...O62	0.94	1.91	2.748(3)	147.7	x, y+1, z
O91-H91...O51	0.95	2.02	2.712(4)	128.1	
O95-H95...O21	0.93	2.22	2.801(4)	119.8	
<b>3H-2((R)- 2-BuOH)</b>					
O2-H2...O21	0.94	1.89	2.791(2)	160.9	x, y+1, z
O21-H21...O81	0.90	2.02	2.882(2)	160.2	x+1, y, z
O32-H32...O9 <sup>i</sup>	0.94	1.86	2.755(3)	159.3	x, y+1, z
O51-H51...O32	0.94	1.84	2.736(2)	159.3	x, y-1, z
O62-H62...O51	0.93	2.02	2.810(3)	141.4	x-1, y-1, z
O81-H81...O91	0.94	1.89	2.779(3)	157.4	x-1, y-1, z
O91-H91...O2	0.93	1.89	2.809(2)	171.1	
O95-H95...O62	0.95	2.02	2.716(3)	128.6	
<b>3H-2((RS)- 2-BuOH)</b>					
O2-H2...O82 <sup>i</sup>	0.83	2.03	2.823(3)	160.3	x-1, y-1, z
O21-H21...O92	0.87	1.98	2.829(5)	168.3	
O32-H32...O51	0.83	2.03	2.797(3)	154.8	x, y-1, z
O51-H51...O21	0.91	2.03	2.874(3)	153.3	x, y+1, z
O62-H62...O100	0.86	1.95	2.721(7)	148.3	
O82-H82...O62	0.86	1.92	2.743(3)	158.7	x+1, y, z
O92-H92...O32	0.84	2.00	2.825(6)	167.2	
O100-H100...O2	0.84	1.89	2.602(7)	141.5	

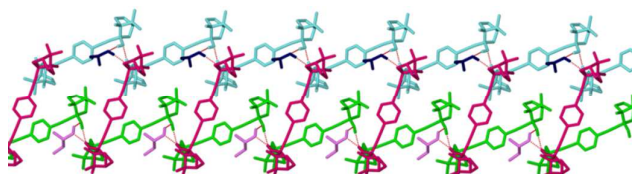


Fig. 4 The molecular arrangement in structures 4-6. The host and guest molecules are linked via hydrogen bonding to form hydrogen bonded tetramers

Inspection of the non-bonded interactions between the disordered guest B in structure 1, shows that the major 72%(S) is hydrogen bonded to the host, while the 28%(R) minor component is not. In structure 2, guest B (57%(S), 43%(R)) is disordered but shares a common position of the guest carbonyl oxygen. This accounts for the smaller difference in the enantiomeric excess between structures 1 and 2.

Fig. 5 (a and b) show the 2D plots generated from the Hirshfeld surface of guest 1 and guest 2 of structure 4 respectively. Since the three structures are isostructural, only the 2D plots of the two guests in structure 4 are shown and those of structures 5 and 6 are given in the ESI (Fig. S1). The spikes labelled 1, 2 and 3 correspond to the O...H, H...H and the C...H interactions respectively. The fingerprint plots for guest 1 and guest 2 are significantly different; guest 1 which is located in voids has a higher percentage of (host)-C...H-(guest) interactions as compared to that of guest 2 which is located in channels. Conversely, there are significantly more (host)-H...H-(guest) interactions between guest 2 and the host than between guest 1 and the host. This trend is also observed in structures 5 and 6 (Fig. S2). Fig. 5(c-d) shows the 2D plots of the Hirshfeld surface generated from the three hosts and two guests in the asymmetric unit for structures 4-6

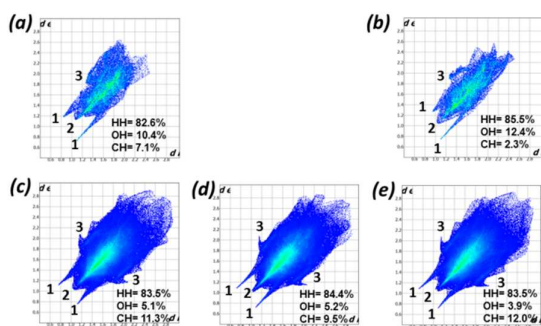


Fig. 5 2D plots for (a) guest 1, (b) guest 2 and (c-e) host and guest in the asymmetric unit of compound **4**, **5** and **6**. Spikes labelled **1**, **2** and **3** correspond to O...H, H...H and C...H interactions respectively

respectively. The 2D plots for all the three structures are similar and, the percentage contributions for the main interactions follow the order H...H>C...H>O...H.

Kalman<sup>18</sup> developed a series of similarity indices which can be employed to measure the extent of isostructurality. The simplest is the unit cell similarity index  $\Pi$ , defined as

$$\Pi = |(a+b+c)/(a'+b'+c') - 1| \approx 0$$

where  $a$ ,  $b$ ,  $c$  and  $a'$ ,  $b'$ ,  $c'$  are the orthogonalised unit cell parameters of the two structures. In our case the  $\Pi$  index for the three structures **4**, **5** and **6**, taken in pairs is always <0.009.

In a previous study,<sup>6a</sup> a similar type of host compound 2,2'-(anthracene-9,10-diyl-diethynylene) diborneol was employed in the resolution of (*RS*)-phenyloxirane. The resultant inclusion compound included only (*S*)-phenyloxirane. These results were attributed to the presence of a 'specificity pocket' within the crystal structure which positions the guest for hydrogen bonding as well as  $\pi\cdots\pi$  interactions.

In the present study, the question that arises is: why does the title host not discriminate between (*R*)- and (*S*)- 2-butanol? We surmise that these two enantiomers are not sufficiently different to act as discriminating templates in the formation of the inclusion compounds and that the host...host recognition, by way of inter-host hydrogen bonds, forms a strong and stable framework in which the guest butanol molecules are accommodated irrespective of their chirality. This is evidenced by the similarity of the metrics of the hydrogen bonds (Table 2) in the three structures, the constancy of the maps of the non-bonded interactions shown in the fingerprint plots (Fig. 5) and in the volumes and topologies of the void guest space which were mapped. A similar effect was noted in the attempted resolution of 2-butylamine by a series of chiral host compounds, derived from tartaric acid, which were used singly or in combination.<sup>19</sup>

## Conclusions

In summary, the host compound 2,2'-(benzene-1,4-diyl-diethynylene)diborneol was crystallised with racemic methylcyclohexanones as well racemic and enantiopure

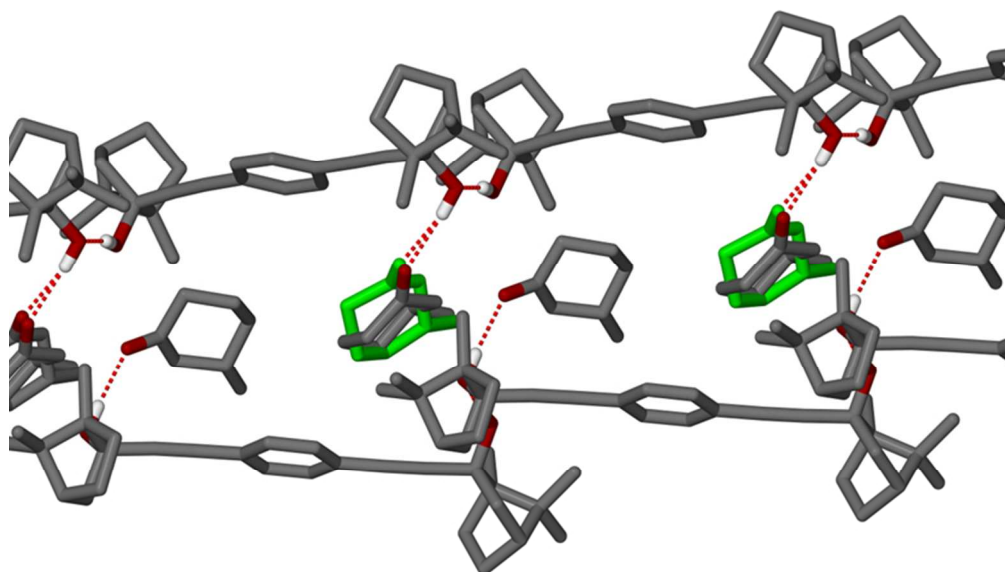
2-butanol. An enantiomeric excess of 72% (*S*) and 57% (*R*) was achieved for 2- and 3-methylcyclohexanones respectively, as determined by crystallographic refinement. The host compound showed no discrimination when crystallised with racemic 2-butanol and the structures with racemic and enantiomerically pure 2-butanol are similar. This is attributed to the size of the guest 2-butanol molecules as well as the strong hydrogen bonds that direct the assembly of the host framework.

## Acknowledgements

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## Notes and references

- E. Weber In: *Inclusion Compounds*; Eds., J. L. Atwood, J.E.D. Davies, D.D. MacNicol, Oxford University Press: Oxford, 1991, vol. 4, pp. 188-262
- (a) R. Bishop, *Chem. Soc. Rev.* 1996, 311; (b) G.R. Desiraju In: *Comprehensive Supramolecular Chemistry*; Eds., D.D. MacNicol, F. Toda, R. Bishop, Elsevier: Oxford, 1996, vol. 6, pp. 1-22
- L. R. Nassimbeni, H. Su and T-L. Curtin, *Chem. Commun.*, 2012, **48**, 8526
- T. Müller, J. Hulliger, W. Seichter, E. Weber, T. Weber and M. Wübhenhorst, *Chem. Eur. J.*, 2000, **6**, 54
- T. Müller, W. Seichter and E. Weber, *New J. Chem.*, 2006, **30**, 751
- (a) P. P. Korkas, E. Weber, M. Czugler, G. J. Náray-Szabó, *Chem. Soc., Chem. Commun.* 1995, 2229; (b) M. Czugler, P. P. Korkas, P. Bombicz, W. Seichter; E. Weber, *Chirality*, 1997, **9**, 203
- E. Weber, P. P. Korkas, M. Czugler and W. Seichter, *Supramol. Chem.* **2004**, **16**, 217
- Bruker 2005, *APEX2*, Version 1.0-27. Bruker AXS Inc., Madison, Wisconsin, USA
- Bruker 2004, *SAINT-Plus* (including *XPREP*), Version 7.12. Bruker AXS Inc., Madison, Wisconsin
- Bruker 2003, *XPREP2*, Version 6.14. Bruker AXS Inc., Madison, Wisconsin, USA
- COLLECT Data Collection Software (1999) Nonius, Delft, The Netherlands
- Otwinowski, Z.; Minor, W. Processing of X-ray diffraction data collected in oscillation mode. *Methods in enzymology, Macromolecular Crystallography, part A*, **1997**, **276**, 307-326
- G. M. Sheldrick, *SHELXS-97* and *SHELXL-97* programs for crystal structure determination and refinement, 1997, University of Göttingen, Germany
- L. J. Barbour, *J. Supramol. Chem.*, 2001, **1**, 189
- M. Lusi and L. J. Barbour, *Cryst. Growth Des.*, 2011, **11**, 5515
- (a) I. J. Bruno, J. C. Cole, P. R. Edgington, M. Kessler, C. F. Macrae, P. McCabe, J. Pearson and R. Taylor, *Acta Crystallogr., Sect. B: Struct. Sci.*, 2002, **58**, 389; (b) C. F. Macrae, I. J. Bruno, J. A. Chisholm, P. R. Edgington, P. McCabe, E. Pidcock, L. Rodriguez-Monge, R. Taylor, J. van de Streek and P. A. Wood, *J. Appl. Crystallogr.*, 2008, **41**, 466; (c) C. F. Macrae, P. R. Edgington, P. McCabe, E. Pidcock, G. P. Shields, R. Taylor, M. Towler and J. van de Streek, *J. Appl. Crystallogr.*, 2006, **39**, 453
- (a) M. A. Spackman and D. Jayatilaka, *CrystEngComm*, 2009, **11**, 19; (b) S. K. Wolff, D. J. Grimwood, J. J. McKinnon, M. J. Turner, D. Jayatilaka and M. A. Spackman, *CrystalExplorer (Version 3.1)*, University of Western Australia, 2010; (c) M. J. Turner, J. J. McKinnon, D. Jayatilaka and M. A. Spackman, *CrystEngComm*, 2011, **13**, 1804
- (a) A. Kálmán, G. Argay, D. Scharfenberg-Pfeiffer, E. Höhne, B. Ribár, *Acta Cryst.*, 1991, **B47**, 68; (b) A. Kálmán, L. Párkányi, G. Argay, *Acta Cryst.*, 1993, **B49**, 1039; (c) L. Fábrián and A. Kálmán, *Acta Cryst.* 1999, **B55**, 1099
- N.B. Bathori and L.R. Nassimbeni, *Cryst. Growth Des.* 2012, **12**, 2501



Partial resolution of racemic methylcyclohexanone and 2-butanol via inclusion with a borneol dumb-bell host